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Effect of mechanical and air-particle cleansing protocols of provisional cement on immediate dentin sealing layer and subsequent adhesion of resin composite cement

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Short title: *Effect of cleansing protocols on immediate dentin sealing and adhesion*

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Abstract: Immediate dentin sealing (IDS) could avoid contamination of dentin from impression material and provisional cement but prior to final cementation of indirect restorations, removal of the provisional cement may damage the IDS. The objectives of this study were to investigate the effect of mechanical and air-particle cleansing protocols of provisional cement on IDS layer and subsequent adhesion of resin composite cement. The cuspal dentin surfaces of human third molars (N=18, $n_{\text{quadrant}}=72$) were exposed by a low speed diamond saw under water cooling and conditioned with an adhesive system based on 3-step etch and rinse technique (OptiBond FL). Provisional cement (Freegenol) was applied on each specimen. They were then randomly divided into 6 subgroups where the provisional cement was removed either by 1) Air-borne particle abrasion with 50 μm Al_2O_3 particles at 2 bar (AL2), 2) Air-borne particle abrasion with 50 μm Al_2O_3 particles at 3.5 bar (AL3.5), 3) Air-borne particle abrasion with 30 μm SiO_2 particles at 2 bar (SL2), 4) Air-borne particle abrasion with 30 μm SiO_2 particles at 3.5 bar (SL3.5), 5) Prophylaxy paste (Cleanic) (PP) or 6) Pumice-water slurry (PW) at 1500 rpm for 15 s. The dentin surface on each tooth was assigned to 4 quadrants and each quadrant received the cleansing methods in a clockwise sequence. The non-contaminated and non-cleansed teeth acted as the control (N=3, $n_{\text{quadrant}}=12$) (C). Two separate teeth, contaminated and cleansed according to 6 cleansing protocols, were allocated for Scanning Electron Microscopy (SEM) analysis (x2000). The dentin surfaces in each quadrant received resin composite luting cement (Variolink II, Ivoclar Vivadent) incrementally in a polyethylene mould (diameter: 1 mm^2 ; height: 4 mm) and photo-polymerized. The specimens were stored in distilled water for 24 hours at 37°C until the testing procedures and then shear force was applied to the adhesive interface until failure occurred in a Universal Testing Machine (0.5 mm/min). Microshear bond (μSBS) was calculated by dividing the maximum load (N) by the bonding surface area of the resin cement. Failure types were analyzed using optical microscope and SEM. Data (MPa) were analyzed using one-way ANOVA ($\alpha=0.05$). Two-parameter Weibull distribution values including the Weibull modulus, scale (m) and shape (θ), values were calculated. Mean μSBS results (MPa) showed significant difference between the experimental groups ($P=0.011$) and were in descending order as follows: C (8 ± 2.3)^a<AL2 (6.7 ± 2.4)^b<PP (6.9 ± 2)^b<PW (6.5 ± 2.1)^b<AL3.5 (5.8 ± 1.1)^b<SL2 (5.3 ± 1)^b<SL3.5 (5.2 ± 1)^b. Failure types were predominantly mixed failure type between the

dentin and the adhesive resin, which is a combination of adhesive and cohesive failure in the adhesive resin. Cohesive failure in the dentin was not observed in any of the groups. Weibull distribution presented lower shape (σ) for C (3.9), AL2 (3.2), PP (3.5) and PW (3.6). SEM analysis showed rough surfaces especially in air-abraded groups whereas mechanical cleansing methods presented smoother surfaces and partially covered by particle remnants all of which occluded the dentin tubuli.

Keywords: Adhesion, air-abrasion, cleansing protocols, contamination, immediate dentin sealing

Introduction

In order to create sufficient space for the indirect fixed-dental-prosthesis (FDP), in clinical practice, most of the time enamel has to be removed with rotating instruments leading to exposure of the dentinal tubules. Dentin exposure may cause bacterial diffusion and trigger pulpal inflammatory response with subsequent formation of reparative dentin [1-3]. In several studies, a significant correlation between microbial microleakage and pulpal inflammation has been demonstrated [3-7]. The rate of bacterial invasion, which is approximately 1.6 $\mu\text{m}/\text{day}$, has been found to increase over time [2]. In a relatively short period of time (up to 4 days), bacteria can infiltrate the tubules. The odontoblastic processes, collagen fibers, the kinetics of tubular fluid and immunological function do not seem to be sufficient to inhibit this process. It is also inevitable that the exposed dentin subjected to mechanical, thermal, tactile or osmotic stimuli results in hypersensitivity [1,8].

Dentin hypersensitivity is inhibited by the precipitation of water-insoluble substances at the orifices of dentinal tubules. One approach for the treatment of hypersensitivity involves sealing of dentinal tubules or application of sedative agents, and promotion of dentin remineralisation [9]. Usually potassium nitrate, oxalate, fluoride and triclosan-based dentin desensitizers are recommended immediately after tooth preparation [10]. The use of such barriers to seal the dentinal tubules prior to cementation has been advocated in order to reduce the effect of external stimuli on hypersensitivity. Some ingredients present in dentin desensitizers may induce chemical interaction with organic substances of the dentin that may consequently affect the sealing and bonding characteristics of the adhesive cement system [9,11-13].

Immediate dentin sealing (IDS) or dual bonding technique has been suggested as an alternative to delayed dentin sealing in order to overcome hypersensitivity and avoid contamination of dentin from impression material and provisional cement [14-17] and at the same time reduce marginal leakage [18-25]. IDS could circumvent contamination of dentin from impression material and provisional cement but in fact clinically prior to final cementation of indirect restorations, removal of the provisional cement may damage the IDS. Cleansing methods used for eliminating provisional cement remnants are principally based on mechanical procedures using rotating instruments with or without slurry of pumice or prophylaxis paste and

different rotation per minute (rpm) or deposition of particles using chairside air-abrasion technologies. Air abrasion protocols require the use of alumina particles or alumina particles coated with silica usually with particle size of up to 50 µm for chairside applications. Alternatively, provisional cement could also be removed with a diamond bur at low speed [21] but may impair the precision of the succeeding FDP. On the other hand, some authors favour the use of airborne alumina particle abrasion claiming that it cleans the surface more homogeneously than pumice [22]. Thus, there seems to be no agreement to date on how to clean the IDS coated dentin surface without impairing adhesion.

The objectives of this study therefore, were to investigate the effect of mechanical and air-particle cleansing protocols of provisional cement on IDS layer and subsequent adhesion of resin composite cement to dentin. The null hypothesis tested was that cleansing regimens used for removal of cement contamination from IDS created dentin surface would not show difference in adhesion of resin composite cement to dentin.

Materials and Methods

Specimen preparation

The types, brands, main chemical compositions, manufacturers and batch numbers of the materials used for the experiments are listed in Table 1. Schematic description of the experimental design is presented in Fig. 1.

Human mandibular wisdom teeth (N=18, $n_{\text{quadrant}}=72$) were used in this study. After tissue remnants were removed with a scaler (H6/H7; Hu-Friedy, Chicago, IL), teeth were stored in 0.5% Chloramin T for 2 weeks. The roots were removed from the coronal parts using a diamond disc (IsoMet 1000, Buehler Ltd, USA) under water-cooling. The coronal part of teeth were embedded in a polyvinyl chloride (PVC) mould with their occlusal surfaces exposed using auto-polymerizing acrylic resin (Scandiquick, Scandia, Hagen, Germany). A low-speed diamond saw (Well Precision Diamond Wire Saw, Norcross, USA) under water-cooling was used to remove the cusps and expose the superficial dentin which was then ground finished using 600, 800 and 1000-grit silicone carbide abrasive papers under water cooling for 5 s in sequence.

Dentin surfaces were conditioned with 3-step etch and rinse adhesive system (OptiBond FL, Kerr, Orange, Calif) according to the manufacturer's recommendations. Dentin surfaces were initially etched for 15 s using 37% H₃PO₄, rinsed for 15 s and gently air-dried for 5 s. First, primer (Bottle 1) was applied with a brush for 15 s, gently air-dried for 5 s, and adhesive resin (Bottle 2) was applied with a brush for 15 s, air thinned for 3 s and photo-polymerized for 30 s using an LED polymerization unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) from a constant distance of 2 mm from the surface. This procedure was considered as creation of the IDS layer. The output of the polymerization unit was 1100 mW/cm² verified by a radiometer (Demetron LC, SDS Kerr, Orange, CA, USA). Dentin surfaces were then coated with eugenol-free provisional cement (Freegenol, GC International, Tokyo, Japan) under a glass slab and stored in distilled water for 24 hours. The non-contaminated and non-cleansed teeth acted as the control (N=3, n_{quadrant}=12).

They were then randomly divided into 6 subgroups (n_{quadrant}=10 per group) where the provisional cement was removed using one of the following cleansing methods:

Cleansing protocols

Group AL2: Provisional cement was removed from dentin surfaces using a chairside air-abrasion device (Ronvig, Daugård, Denmark) where air-borne particle abrasion was achieved using 50 µm Al₂O₃ particles (Korox, Bego, Bremen, Germany) at 2 bar for 5 s from a distance of approximately 10 mm in rotating motions.

Group AL3.5: Air-borne particle abrasion was performed as in Group AL2 but at 3.5 bar.

Group SL2: In this group, air-borne particle abrasion was achieved with 30 µm SiO₂ particles (CoJet Sand, 3M ESPE, Seefeld, Germany) at 2 bar.

Group SL3.5: In this group, air-borne particle abrasion was identical as in Group SL2 but at 3.5 bar.

Group PP: Provisional cement was removed with prophylaxy paste (Cleanic, Kerr, Orange, CA) using rotary nylon brush at 1500 rpm for 15 s.

Group PW: In this group pumice-water slurry was applied using rotary nylon brush at 1500 rpm for 15 s.

The dentin surface on each tooth was assigned to 4 quadrants and each quadrant received the cleansing methods in a clockwise sequence (Fig. 2). On each tooth, two lines were drawn mesiodistally and buccolingually to divide the bonding area into 4 quadrants. During cleansing of one quadrant, the remaining quadrants were protected with a teflon shield. After cleansing procedures, IDS was created again as described above.

One operator performed all cleansing and bonding procedures. The specimens were then stored in distilled water for 24 hours at 37°C.

Resin composite luting cement (Variolink II, Ivoclar Vivadent) was condensed into polyethylene moulds (inner surface area 1 mm²; height: 4 mm) in two increments. Each layer was photo polymerized for 40 s using an LED polymerization unit (Bluephase, Ivoclar Vivadent) from a constant distance of 2 mm from the surface. The specimens were then stored in distilled water for 24 hours at 37°C until experiments.

Microshear (μ SBS) test

Specimens were mounted in the jig of the Universal Testing Machine (Z010, Zwick, Ulm, Germany) and shear force was applied to the adhesive interface until failure occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 0.5 mm/min. μ SBS (MPa) was calculated by dividing the maximum load (N) by the bonding surface area of the resin cement (mm²).

Failure analysis and microscopy evaluation

Failure sites were initially observed using an optical microscope (Zeiss Supra V50, Carl Zeiss, Oberkochen, Germany) and classified as follows: Type I: Adhesive failure between the adhesive resin and the dentin; Type IIa: Mixed failure between the adhesive resin and the dentin with less than half of the adhesive remained on the dentin surface; Type IIb: Mixed failure between the adhesive resin and the dentin with more than half of the adhesive resin remained on the dentin surface; Type III: Cohesive failure in the cement; Type IV: Cohesive failure in the dentin.

Additionally, in order to observe the structural changes on the dentin or IDS, after cleansing methods, two further specimens from each group were first sputter-coated with a 3 nm thick layer of gold (80%) ,

palladium (20%) (90 s, 45mA; Balzers SCD 030, Balzers, Liechtenstein) and analyzed using cold field emission Scanning Electron Microscope (SEM) (LEO 440, Electron Microscopy Ltd, Cambridge, UK). Images were made at 25 kV at a magnification of x200 to x2000.

Statistical analysis

A sample size of 10 in each group was calculated to have more than 80% power to detect a difference of 5 MPa between the mean values. According to the two-group Satterthwaite t-test (SPSS Software V.20, Chicago, IL, USA) with a 0.05 two-sided significance level, this assumes that for conditioning with Al₂O₃ standard deviation is 5.38 and with SiO₂ standard deviation is 5.02. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data (MPa) were normally distributed, 1-way analysis of variance (ANOVA) were applied to analyse possible differences between the groups. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution, including the Weibull modulus, scale (m) and shape (θ), to interpret predictability and reliability of adhesion (Minitab Software V.16, State College, PA, USA). $P < 0.05$ was considered to be statistically significant in all tests.

Results

Mean μ SBS results (MPa) showed significant difference between the experimental groups ($P=0.011$). All contaminated and cleaned groups showed significantly lower results compared to that of the control group (C) and were in descending order as follows: C (8 ± 2.3)^a < AL2 (6.7 ± 2.4)^b < PP (6.9 ± 2)^b < PW (6.5 ± 2.1)^b < AL3.5 (5.8 ± 1.1)^b < SL2 (5.3 ± 1)^b < SL3.5 (5.2 ± 1)^b (Table 2).

Weibull distribution presented lower shape (θ) for C (3.9), AL2 (3.2), PP (3.5) and PW (3.6) (Fig. 3).

Failure types were predominantly mixed failure type between the dentin and the adhesive resin, which is a combination of adhesive and cohesive failure in the adhesive resin (Fig. 4). Cohesive failure in the dentin was not observed in any of the groups.

SEM analysis showed rough surfaces especially in air-abraded groups whereas mechanical cleansing methods presented smoother surfaces and partially covered by particles all of which occluded the dentin tubuli (Figs. 5a-f).

Discussion

An effective and durable cementation protocol is crucial to increase service life of debonded FDPs where temporary cement remnants need to be removed. This becomes more of a subject in minimal invasive reconstructions where the retention relies solely on adhesion of the cement to the dental tissues and the restorative material. Contamination of dentin surfaces during the whole restorative or prosthetic workflow from preparation of the tooth to impression making and subsequently provisional cementation may hinder adhesion of the final cement layer to dentin. Hence, dentin surfaces needs to be cleaned from such contaminants prior to final cementation, which could be accomplished using different methods and may have impact at different levels on the IDS that serves for protecting the dentin surfaces from contamination and at the same time decrease hypersensitivity. For this reason, this study was undertaken to investigate the effect of mechanical and air-particle cleansing protocols of provisional cement on IDS layer and subsequent adhesion of resin composite cement to dentin. Based on the results of this study, since there was no significant difference between the cleansing regimens in terms of adhesive strength, the null hypothesis that the cleansing regimens would not show significant difference on the adhesion of resin composite cement to dentin, could be accepted.

Several testing methodologies, (i.e. macroshear, microshear, macrotensile, and microtensile tests) have been suggested for evaluation of the bond strength of resin-based materials to dentin. Accordingly, in order to measure the bond strength values between an adherent and a substrate accurately, it is crucial that the bonding interface should be the most stressed region, regardless of the test methodology being employed [26]. In this study, bond strength was tested using μ SBS test. With this method, inherent problems associated with macroshear test and microtensile tests could be eliminated. While the macroshear test results in cohesive failure of the substrate, not revealing the true bond strength, pre-test failures or misalignment of the specimens are the other problems associated with microtensile test [26]. In μ SBS test, bonded cylindrical resin cement surface is small enough not to be negatively affected from such factors yielding to more reliable results. One translucent polyethylene mould filled with the cement was bonded on

each dentin specimen surface. Through clockwise application of the 6 cleansing regimens in 4 quadrants of each tooth, the possible effect of variation in dentin tubule orientation on the adhesion was avoided.

In this study, IDS was created using 3-step “etch and rinse” adhesive. Such adhesive systems require a moist substrate for optimal bonding [27], making it highly sensitive since the collapse of over-dried, exposed collagen acts as a difficult substrate for the monomer infiltration. However, this also indicates that in the case of increased dentin wetness, which occurs when dentin depth increases, an overly wet condition may be created [27]. This makes 3-step “etch and rinse” adhesives more technique sensitive compared to self-etch adhesive types. The substrate on which IDS was studied in this investigation was superficial dentin. It has been previously reported that superficial dentin is a favourable substrate for the “etch and rinse” system compared to the deep dentin [28].

A generalized protocol about the elimination of cement remnants from dentin surface to date is lacking. Cleansing protocols for removal of provisional cements has been studied [29] and categorized into mechanical and chemical methods [29]. Mechanical cleansing protocols included the use of pumice aided with rotary instrumentation, air polishing and air-abrasion with abrasive particles, the use of excavator, air-scaler and sonic toothbrush [30]. On the other hand, chlorhexidine digluconate, sodium hypochloride, ethanol containing agents, hydrogen peroxide and polyacrylic acid applications have been proposed as chemical cleansing agents. One of the most common and cheapest medium to remove cement remnants from tooth surface is pumice-water slurry. Considering possible impairment in precision of the reconstruction, multiple mechanical cleansing protocols such as composite finishing burs, tungsten carbide burs and ultrasonic scaler tip were not tested for removal of provisional cement.

In this study, both cleansing with prophylaxy paste and pumice-water slurry performed similar in terms of mean bond strength to dentin coated with IDS. Moreover, the results were also not significant from those of other air-abrasion protocols where the latter require utilizing a chairside air-abrasion device in addition to the regular armamentarium in a clinical setting. However, when Weibull distribution is considered, both mechanical cleansing methods provided less reliable adhesion results as opposed to other methods based on air-abrasion. Similarly, Santos et al. have reported negative effect of the use of pumice on the bond

strength of the resin to fresh dentin due to obscuration of the dentinal tubule openings by pumice residues [31]. Thus, the use of air-abrasion still seems to be more beneficial to optimize adhesion of resin cement to IDS. The duration of mechanical cleaning was limited to 15 s under 1500 rpm. Prolonged duration of mechanical cleaning could either smear the provisional cement further in the IDS, completely remove the IDS layer or create heat transmission to dentin due to prolonged friction. For this reason, and also considering the small working environment in each 4 quadrants of teeth, short application duration was employed.

In the present study, pumice-water slurry was applied on the provisional cement surface where the dentinal tubules were already impregnated by the resin. Since dentin tubuli exposure could not be observed clinically, in this study after cleansing procedures, an attempt was made to create the IDS again. Possibly, reactivation of the surface with an adhesive resin, bearing in mind that the cement remnants were not completely removed or partially smeared on the IDS, have improved the covalent bonding between the adhesive resin and the methacrylate groups of the resin luting cement. The obtained results were therefore a kind of resin-resin bond. On the other hand, surface roughness of the existing composites and available free monomers, activated by a bonding agent increased the bond strength in composite repair studies [32]. This could have increased the results but clinically this attempt would interfere with the fit of the restorations, which needs to be further investigated.

While the Weibull parameters were not favourable for AL2 under the same conditions, Groups AL3.5, SL2 and SL3.5 presented more reliable results. This could be attributed to the variations in the morphology of the particles in that, 30 μm alumina particles coated with silica has been reported to embed on the surface better than the alumina particles with more coarse morphology that eventually bounces back after deposition on a given substrate [33]. Deposition parameters associated with particle deposition techniques certainly have an impact on the results [34] and this seems to be more essential for Al_2O_3 particles since increased pressure from 2 to 3.5 bar may increase the surface roughness [35] with this sand particle and increase the reliability of adhesion to IDS.

Interpretation of μ SBS results should be accompanied with failure type analysis. Failure types were predominantly mixed failure type between the dentin and the adhesive resin, which is a combination of adhesive and cohesive failure in the adhesive resin at varying degrees. Thus, the adhesion of the IDS does not seem to be ideal. Although not frequent, except in groups SL2 and SL3.5, cohesive failures in the cement were noted. This could be due to better adhesion of the free monomers available on the IDS surface with those of the resin cement. One explanation for the lack of cohesive failures in SL groups could be due to better penetration, increased surface roughness but in turn removal of oxygen inhibited layer that eventually yields to less adhesive debonding between the IDS and the resin cement [36]. Yet, the bond strength results cannot verify this assumption. The lack of cohesive failure in the dentin in any of the groups questions the clinical reliability of the adhesion overall to dentin. SEM analysis demonstrated rough surfaces especially in air-abraded groups that could also affect the wettability of the IDS surface. On the other hand, mechanical cleansing methods presented smoother surfaces and with some evident particles all of which apparently occluded the dentin tubuli.

As the specimens were tested only after 24 hours water storage, this study simulates an early recementation scenario. Maximum polymerization with these cements may take up to 24 hours [36] and during this time the patients need to function and consequently early debondings may occur. The extended storage time in water or challenging the interfaces in thermocycling after initial cementation could be taken into account in future studies. However, it has to be noted that during thermocycling process, with some cement systems further polymerization and thereby increased degree of conversion could be observed. For this reason, short and long-term aging in the same study may bring additional information on the adhesion behaviour of resin composite cements to IDS.

Briefly, considering adhesion results, failure types and Weibull parameters, provisional cement remnants on the IDS could best be removed by cleansing methods using air-abrasion methods for a short duration of 5 s per quadrant on a tooth surface that needs to be reactivated by adhesive resin after cleansing.

Conclusions

From this study, the following could be concluded:

1. All mechanical and air-particle cleansing protocols of provisional cement on immediate dentin sealing layer performed similar in terms of adhesion of resin luting cement on dentin.
2. Considering Weibull parameters, characteristics of adhesion seems to be less reliable for air-abrasion with 50 μm Al_2O_3 particles at 2 bar and mechanical cleansing methods with prophylaxy paste or pumice water slurry, indicating the need for some pressure to remove the provisional cement from dentin surfaces.
3. Failure types were predominantly mixed failure type between the dentin and the adhesive resin, which is a combination of adhesive and cohesive failure in the adhesive resin. Cohesive failure in the dentin was not observed in any of the groups.

Clinical Relevance

All mechanical and air-particle cleansing protocols of provisional cement on immediate dentin sealing layer performed similar in terms of adhesion of resin luting cement on dentin providing that according to Weibull parameters, bond reliability was lower for A2, PP and PW.

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Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

References

1. Al-Salehi SK, Burke FJT. Methods used in dentin bonding tests: an analysis of 50 investigations on bond strength. *Quintessence Int* 1997;28:717-723.
2. Brannström M. Reducing the risk of sensitivity and pulpal complications after the placement of crowns and fixed partial dentures. *Quintessence Int* 1996;27:673-678.
3. Brannström M. Infection beneath composite resin restorations: Can it be avoided? *Oper Dent* 1987;12:158-63.
4. Brannström M. The cause of postoperative sensitivity and its prevention. *J Endod* 1986;10:475-481.
5. Christoffersen J, Christoffersen MR, Arends J, Leonardsen ES. Formation of phosphate-containing calcium fluoride at the expense of enamel, hydroxyapatite and fluorapatite. *Caries Res* 1995; 29:223-230.
6. Cardoso PE, Sadek FT, Goracci C, Ferrari M. Adhesion testing with the microtensile method: effects of dental substrate and adhesive system on bond strength measurements. *J Adhes Dent* 2002;4:291-297.
7. Clelland NL, Ramirez A, Katsube N, Seghi RR. Influence of bond quality on failure load of leucite- and lithia disilicate-based ceramics. *J Prosthet Dent* 2007;97:18-24
8. Dagostin A, Ferrari M. Effect of resins sealing of dentin on the bond strength of ceramic restorations. *Dent Mater* 2002;18:304-310.
9. El Zohairy AA, De Gee AJ, Mohsen MM, Feilzer AJ. Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks. *Dent Mater* 2003;19:575-583.
10. Featherstone JD, Glena R, Shariati M, Shields CP. Dependence of in vitro demineralization of apatite and remineralization of dental enamel on fluoride concentration. *J Dent Res* 1990;69:620-625.
11. Gwinnett AJ. Effect of cavity disinfection on bond strength to dentin. *J Esthet Dent* 1992;4:11-13.
12. Gultz J, Do L, Boylan R, Kaim J, Scherer. Antimicrobial activity of cavity disinfectants. *Gen Dent* 1999;47:187-190.
13. Gürgen S, Bolay S, Kiremitçi A. Effect of disinfectant application methods on the bond strength of composite to dentin. *J Oral Rehabil* 1999;26:836-840.

14. Pashley EL, Comer RW, Simpson MD, Homer JA, Pashley DH, Caughman WF. Dentin permeability: sealing the dentin in crown preparations. *Oper Dent* 1992;17:13-20.
15. Paul SJ, Schaerer P. Effect of provisional cements on the bond strength of various bonding systems on dentine. *J Oral Rehabil* 1997;24:8-14.
16. Paul SJ, Schaerer P. The dual bonding technique: a modified method to improve adhesive luting procedures. *Int J Periodontics Restorative Dent* 1997;17:536-545.
17. Magne P, Kim TH, Cascione D, Donovan TE. Immediate dentin sealing improves bond strengths of indirect restorations. *J Prosthet Dent* 2005;94:511-519.
18. Hanssen EK, Asmussen E. Influence of temporary filling materials on effect of dentin-bonding agents. *Scand J Dent Res* 1987;95:516-520.
19. Bertschinger C, Paul SJ, Lüthy H, Schärer P. Dual application of dentin bonding agents: effect on bond strength. *Am J Dent* 1996;9:115-119.
20. Magne P, Belser U. Immediate dentin bonding. In: Magne P, Belser U, eds. *Bonded Porcelain Restorations in the Anterior Dentition-A Biomimetic Approach*. Chicago: Quintessence Publishing Co., 2002:270-273,358-363.
21. Magne P. Immediate dentin sealing: A fundamental procedure for indirect bonded restorations. *J Esthet Restor Dent* 2005;17:144-154.
22. Stavridkis M.M, Krejci I, Magne P. Immediate dentin sealing of onlay preparations: thickness of pre-cured dentin bonding agent and effect of surface cleaning. *Oper Dent* 2005;30:747-757.
23. Magne P, So WS, Cascione D. Immediate dentin sealing supports delayed restoration placement. *J Prosthet Dent* 2007;98:166-174.
24. Duarte S, Buanain de Freitas CR, Saad JR, Sadan A. The effect of immediate dentin sealing on the marginal adaptation and bond strengths of total-etch and self etch adhesives. *J Prosthet Dent* 2009;102:1-9.
25. Hu J, Zhu Q. Effect of immediate dentin sealing on preventive treatment for postcementation hypersensitivity. *Int J Prosthodont* 2010;23:49-52.

26. Della Bona A, Van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. *J Dent Res* 1995;74:1591-1596.
27. Pashley EL, Zhang Y, Lockwood PE, Rueggeberg FA, Pashley DH. Effects of HEMA on water evaporation from water-HEMA mixtures. *Dent Mater* 1998;14:6-10.
28. Özcan M, Mese A. Adhesion of conventional and simplified resin-based luting cements to superficial and deep dentin. *Clin Oral Investig* 2012;16:1081-1088.
29. Sarac D, Sarac YS, Kulunk S, Kulunk T. Effect of the dentin cleansing techniques on dentin wetting and on the bond strength of a resin luting agent. *J Prosthet Dent* 2005;94:363-369.
30. Kanakuri K, Kawamoto Y, Matsumura H. Influence of temporary cement remnant and surface cleaning method on bond strength to dentin of a composite luting system. *J Oral Sci* 2005;47:9-13.
31. Santos-Daroz CB, Oliveira MT, Goes MF, Nikaido T, Tagami J, Giannini M. Bond strength of a resin cement to dentin using the resin coating technique. *Braz Oral Res* 2008;22:198-204.
32. Brendeke J, Özcan M. Effect of physicochemical aging conditions on the composite-composite repair bond strength. *J Adhes Dent* 2007;9:399-406.
33. Özcan M. Airborne particle abrasion of zirconia fixed dental prostheses. *J Esthet Restor Dent* 2014;26:359-362.
34. Özcan M, Raadschelders J, Vallittu P, Lassilla L. Effect of particle deposition parameters on silica coating of zirconia using a chairside air-abrasion device. *J Adhes Dent* 2013;15:211-214.
35. Özcan M, Barbosa SH, Melo RM, Galhano GA, Bottino MA. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. *Dent Mater* 2007;23:1276-1282.
36. Vrochari AD, Eliades G, Hellwig E, Wrbas KT. Curing efficiency of four self-etching, self-adhesive resin cements. *Dent Mater* 2009;25:1104-1108.

Captions to tables and figures:

Tables:

Table 1. The brands, chemical compositions, manufacturers and batch numbers of the materials used for the experiments. bis-GMA: Bisphenol A glycol dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol A glycol dimethacrylate; UDMA: Urethane dimethacrylate; HEMA: Hydroxyethyl methacrylate; MMA: Methylmethacrylate; PMMA: Polymethylmethacrylate; GPDM; Glycerolphosphate dimethacrylate; PAMM: phthalic acid monoethyl methacrylate.

Table 2. Microshear (μ SBS) bond strengths (Mean \pm standard deviation) of resin composite cement on dentin after cleansing methods, maximum, minimum and Confidence Intervals (95%). Same upper-case letters in each column indicate no significant differences ($p>0.05$).

Figures:

Fig. 1. Flow-chart showing experimental sequence and allocation of groups.

Fig. 2 Sketch of the sequence of cleansing regimens applied in 4 quadrants of superficial occlusal dentin clockwise. 1: AL2; 2: AL3.5; 3: SL2; 4: SL3.5; 5: PO; 6: PW. For group abbreviations see Fig. 1.

Fig. 3 Probability plot with Weibull curves (95% CI) using maximum likelihood estimation, scale and shape values for all groups.

Fig. 4 Frequencies of failure modes in percentages. Type I: Adhesive failure between the adhesive resin and the dentin; Type IIa: Mixed failure between the adhesive resin and the dentin with less than half of the adhesive remained on the dentin surface; Type IIb: Mixed failure between the adhesive resin and the dentin with more than half of the adhesive resin remained on the dentin surface; Type III: Cohesive failure in the cement; Type IV: Cohesive failure in the dentin.

Figs. 5a-f SEM images of **a)** AL2, **b)** AL3.5, **c)** SL2, **d)** SL3.5, **e)** PP, **f)** PW at x2000. Note the rougher surfaces especially in air-abraded groups (AL2, AL3.5, SL2, SL3.5) and smoother surfaces with

mechanical cleansing methods where some particles partially remained on the surface and that the dentin tubuli were occluded after all cleansing methods.

Tables:

Materials	Chemical Composition	Manufacturer	Batch Number
Ultra-etch	37.5% H ₃ PO ₄ , silica thickener	Ultradent, South South Jordan Utah, USA	B9BM5
OptiBond FL	Primer: HEMA, GPDM, PAMM, ethanol, water, photo-initiator Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photo initiator	Kerr, Orange, CA, USA	5034497
Freegenol	Base: Zinc oxide, Olive oil, Vaseline, Pigment Accelerator: Polymerized fatty acids, Ester gum, Beewax, Oleic acid	GC international, Tokyo, Japan	1311011
Cobra	50 µm AlO ₂	Renfert, Hilzingen, Germany	1554775
CoJet	30 µm SiO ₂	3M ESPE, Minnesota, USA	542149
Cleanic Prophy Paste	Glycerin, Titaniumdioxide, Ethanol	Kerr	5315565
Pumice	Pumice	Unor, Schlieren, Switzerland	3221580
Variolink II (Low-viscosity)	Monomer Matrix: bis-GMA, UDMA, TEGDMA Inorganic Fillers: Barium Glass, Ytterbium Trifluoride, Ba-Al-fluorosilicate glass, Spheroid mixed oxide Catalyst, stabilizers, pigments	Ivoclar Vivadent, Schaan, Liechtenstein	S47800

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Experimental Groups	n _q	Mean (SD)	Minimum	Maximum	Confidence Interval	
					Lower Bound	Upper Bound
C	12	8±2.3 ^a	5.2	12	-1.0487	3.6723
AL2	12	6.7±2.4 ^b	3.99	11.05	-3.6723	1.0487
AL3.5	12	5.8±1.1 ^b	3.45	7.63	-4.4793	0.2417
SL2	12	5.3±1 ^b	3.57	6.41	-4.9823	-0.2613
SL3.5	12	5.2±1 ^b	3.65	6.5	-5.0383	-0.3173
PP	12	6.9±2 ^b	4.74	11.85	-3.3963	1.3247
PW	12	6.5±2.1 ^b	3.14	9.72	-3.8353	0.8867

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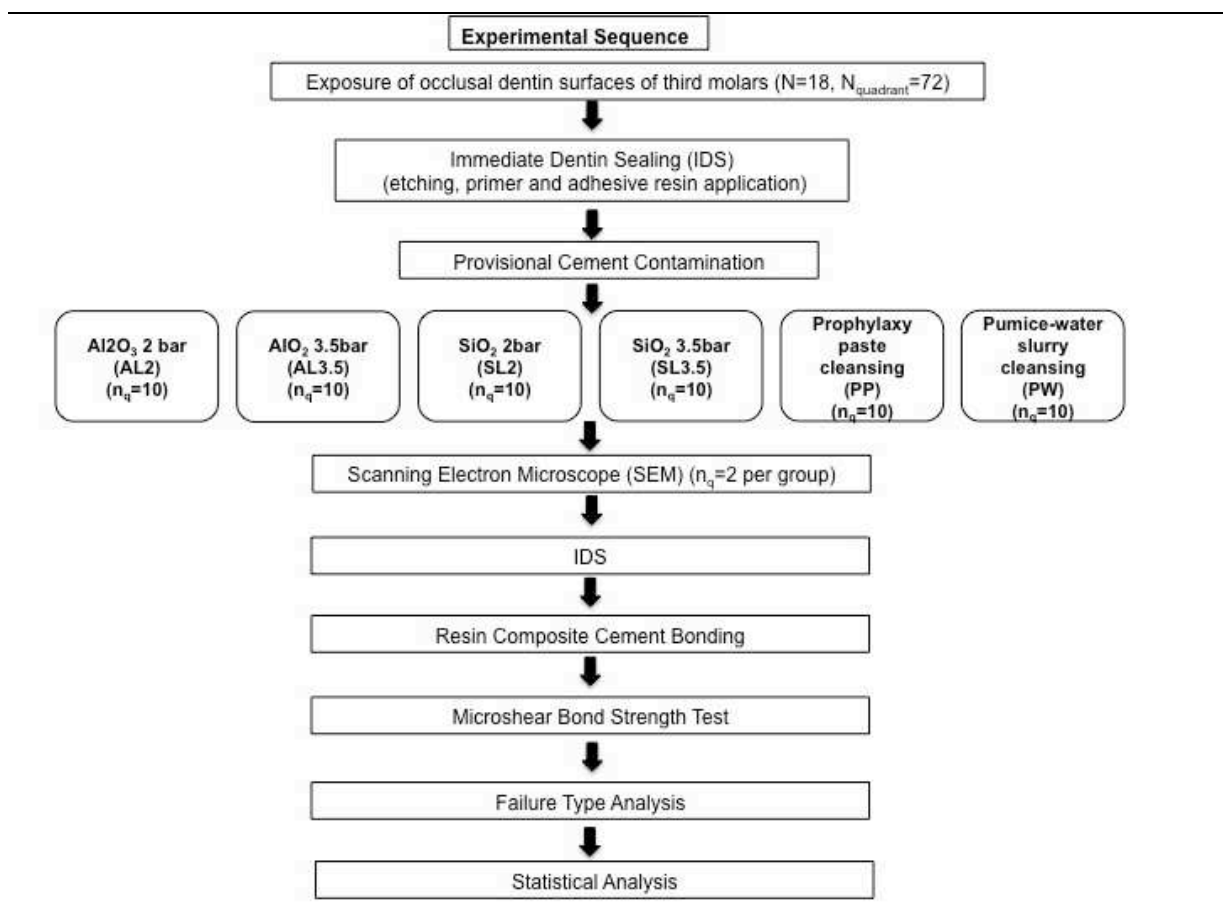


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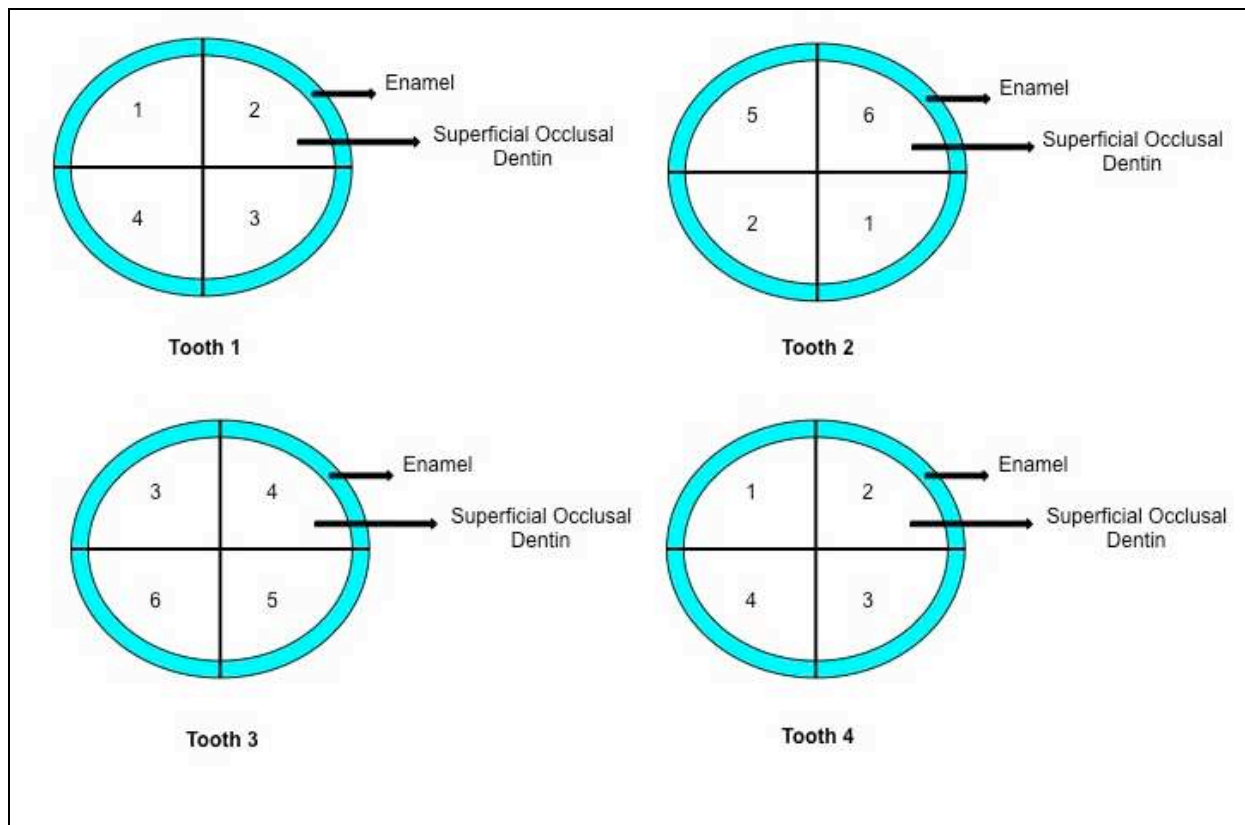


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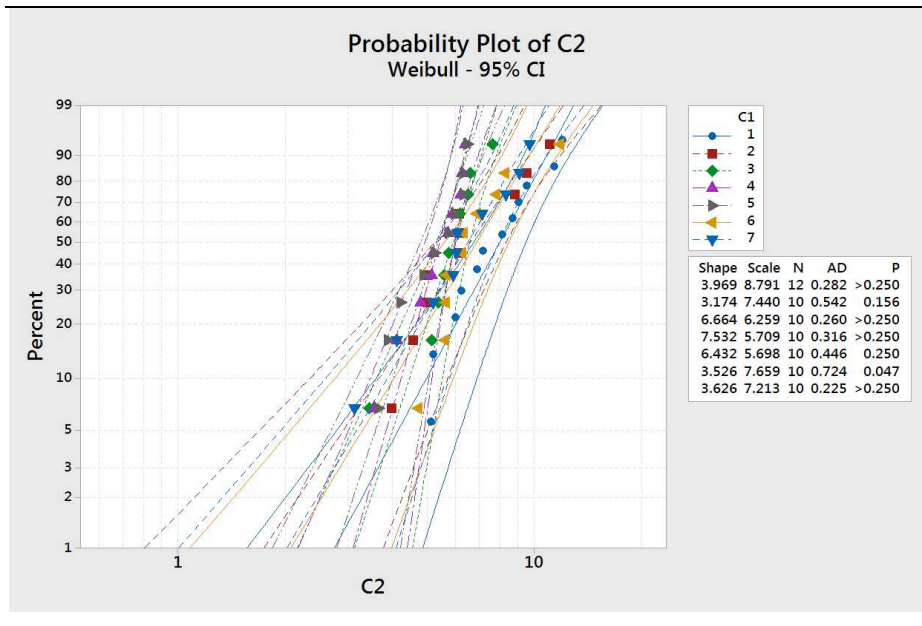


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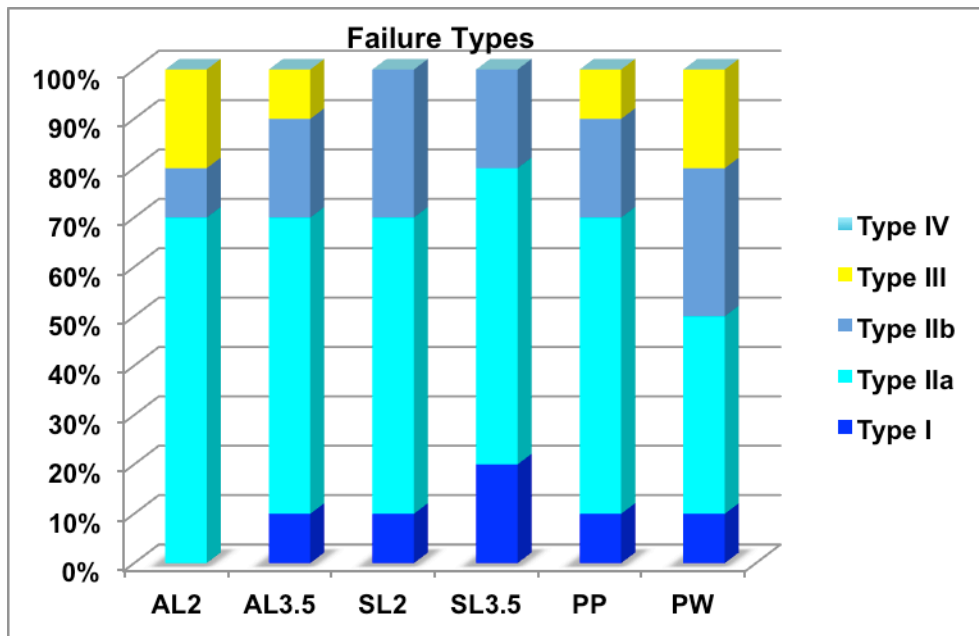
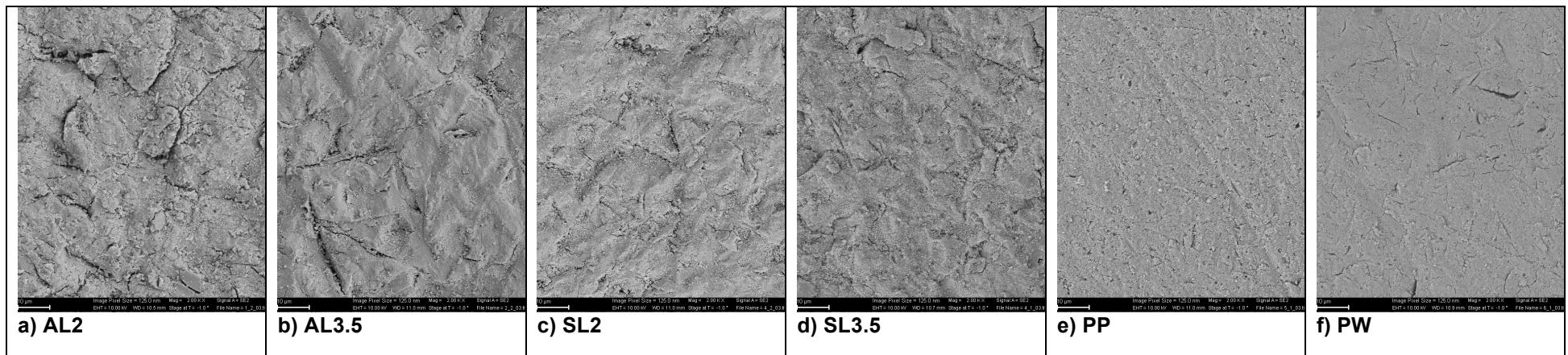


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